CENTRAL INTELLIGENCE AGENCY

INFORMATION REPORT

SECRET
SECURITY INFORMATION

COUNTRY	USSR (Gorkiy Oblast)	REPORT	
SUBJECT	Research Work on Rocket Propellants by German Scientists at the OKA Chemical Plant in Dzerzhinsk	DATE DISTR.	13 May 1953 25X1
		NO. OF PAGES	11
DATE OF INFO.		REQUIREMENT NO.	RD
PLACE ACQUIRED		REFERENCES	25X1

This is UNEVALUATED Information

25X1

THE SOURCE EVALUATIONS IN THIS REPORT ARE DEFINITIVE.
THE APPRAISAL OF CONTENT IS TENTATIVE.
(FOR KEY SEE REVERSE)

1. The OKA Chemical Plant is located in Dzerzhinsk (N56-15, E43-24), nine km east of the main railroad station, and about 240 meters south of the Dzerzhinsk-Gorkiy (N46-20, E44-00) railroad line, just east of Zavod No 96. 25X1

2. Although the official name of the plant was Khimicheskiy Zavod OKA, the numerical designation 365 was still on stamps and certificates and on books in the plant library.

3. The plant had a spur track to the Igumnovo railroad station (N56-16, E43-37), which was northwest of the plant. 25X1 Construction work was still under way in the southern section of the plant area in early 1951. Electric power and steam for the plant were supplied by the neighboring steam-operated power plant (TEZ).

4. The German specialists in the plant worked in what was called the Special Technical Office, which consisted of two rooms of 50 square meters each, one 40-square-meter room for mechanical work, one room for physico-chemical work, one dark room, and one weighing room. The rooms were not equipped until the German specialists arrived with German-made equipment.

5. The six German specialists who worked at the Special Technical Office of the OKA Plant were subordinate to Zavod No 94 in Moscow, near the southern harbor of Moscow, and to the Ministry for Chemical Industry. They received their orders directly from the Ministry. They were assisted by eight technicians and women laboratory workers, two locksmiths, four unskilled workers, and one

SECRET

STATE	ARMY	NAVY	AIR Ev	FBI	AEC	OSI Ev	ATIC Ev	
x	x	x	x			x	x	25X1

(Note: Washington Distribution Indicated By "X"; Field Distribution By "#".)

25 YEAR RE-REVIEW

glass blower, who was an MVD agent and who was assigned to watch the Germans during working hours. All products resulting from experiments of the Special Technical Office were forwarded to Zavod No 94 in Moscow for examination. The German specialists were not informed as to the results of their work or the experimental tests carried out in Moscow. A number of suggestions for new methods which they submitted were refused on the grounds that the basic raw materials required were not available in the USSR. In most cases, work in the Special Technical Office was not connected with the production of the OKA plant. The only exceptions were temporary research work on diphenylguanidine (HN = C $(NH-C_6H_5)_2$) for the OKA Plant and on methacrylic acid ($CH_2 = C(CH_3) - COOH$) for the Rulon Plant in Dzerzhinsk.

6. Soviet personnel of the OKA Plant who were in contact with the Germans during the period from July 1948 to May 1951 included the following: Manager Grigorev (fnu), a chemist; chief engineer Ivanov (fnu); Penkov (fnu), head of Section I (Political Section) and deputy commissar; and Bobyshev (fnu), graduate chemist and manager of the Special Technical Office from 1948 to October 1949. In 1945 and 1946 Bobyshev was in Leuna, Germany, and, in October 1949, he was transferred to Moscow as head manager of the laboratory of Zavod No 94. Shestakov (fnu) was senior laboratory assistant and deputy manager of the Special Technical Office from October 1949 to March 1950. He was followed by Shishov (fnu), who remained until the German experts left the plant. Bukholding (fnu) was economic manager; Genadiy Sergeyevich Sverzidaskiy was the interpreter at the OKA Plant; and Lofshin (fnu) served as the interpreter at the settlement where the German specialists lived. The Special Technical Office at the OKA Plant was periodically inspected by a Soviet general who apparently was a specialist.

[redacted] 25X1

[redacted] Based on the number of workers' passes checked by the gate guard and at the entrance building, about 700 persons, working in three shifts, were employed in the OKA Plant. 25X1

7. During the period from fall 1948 to October 1950, after finishing work on propane oxidation which had been almost completed at the Karpov Institute in Moscow,[1] the German specialists of the Special Technical Office at the OKA Plant primarily did research work on rocket propellants, based chiefly on amines, with nitric acid as an oxidizing agent. Mercaptans, undiluted or diluted as much as 60 percent with gasoline, were later used as fuel.

8. The research work in the field of amines was a continuation of the same work the German group had done rather successfully at the Karpov Institute in Moscow. They concentrated on acetonine ($C_9H_{18}N_2$), a combination which was obtained chiefly by gradually saturating acetone (CH_3COOH_3) with ammonia. Ammonium nitrate was used as a catalyst in this process, with about one part ammonium nitrate being used to about 80 parts acetone. After previous treatment with a caustic soda solution, the solution obtained was dried with solid caustic soda. The acetonine was formulated $C_9H_{18}N_2$ as 2, 2, 4, 6, 6 pentamethyl -1, 2, 5, 6 tetrahydropyrimidine. Other forms of acetonine obtained by treating acetonine with sodium in an alcoholic solution were even better than this acetonine, which showed good hypergolic properties. These were, for example, 2, 4-diamino-N_4 - isopropyl - 2 - methylpentane and 2, 4 - diamino-2-methylpentane.

9. The criterion, indicating whether the agents tested were suited for rocket propellants, was the ignition-delay time required for igniting the agent after being mixed with nitric acid. One successful method of reducing the ignition-delay time was the adding of butyrate of iron to the amine solution as a catalyzer. However, it appeared that, after being stored for a period of two or three months, the solutions treated with this catalyzer formed solid ferruginous deposits. The OKA Plant, therefore, initiated experiments with the objective of using ferruginous nitric acid for igniting the propelling charge. As crystalized nitrate of iron ($Fe(NO_3)_3-9H_2O$) is insoluble in high-concentrated nitric acid, metallic iron was dissolved in this acid. The resulting reaction was violent, making, for example, it possible to add only portions of from 20 to 50 grams, at most, to ten liters of acid at intervals of ten minutes. When this iron solution was distilled off,

a brown paste remained and could again be dissolved easily in high-concentrated nitric acid. The amount of iron (borings, etc.) to be used was 0.5 parts of Fe per 100 parts of nitric acid. Higher percentages of iron failed to increase the reactivity.

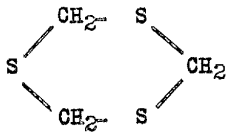
10. Other experiments were made on organic sulphur compounds in amine solutions. Trithioacetone was the first to be tested. The pure substance showed no hypergolic qualities, but it considerably shortened the ignition - delay time as an admixture, up to ten percent, to the acetonine-benzine mixture. This effect was called "promoting effect". The next step was the addition of hydrogen sulfide to acetonine, resulting in a usable mixture, designated KS, which showed definitely improved qualities. The hydrogen sulfide was added to the acetonine until the liquid was entirely interspersed with ammonium sulfide crystals (NH_4HS).[2] The liquid was then sucked off and the filtrate was shortly heated to a temperature of 70°C. Distillative dressing showed that such a mixture consisted of 60 to 70 percent acetonine, while the remaining substance was composed of organic sulfo-nitric compounds which were not determined in detail.

11. Beginning in 1949, the experts in Dzerzhinsk worked with a mixture consisting of 24 percent acetonine, 12 percent aniline ($C_6H_5-NH_2$), 4 percent sulphur, and 60 percent cracked benzine, from which a dark brown solution of unknown composition was obtained. This mixture, like all previous solutions containing acetonine, proved unstable, showing a tar-like precipitation after a storage period of one month. It was possible to delay considerably, but not to stop entirely, these precipitations by adding oxide-free metallic iron powder.

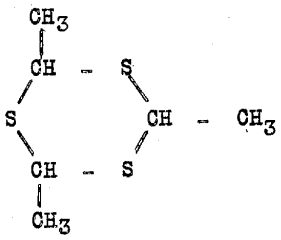
12. In order to reduce to system the obscure hypergolic effects of sulphur compounds, a series of these compounds, of an exactly defined composition, was synthetically prepared and tested, as follows:

Carbon disulfide CS_2

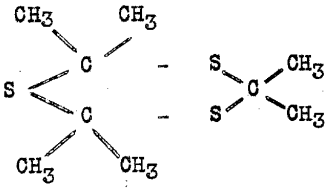
Trithioformaldehyde



Trithioacetaldehyde

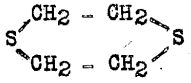


Trithioacetone

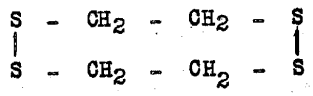


Ethylendimercaptane $HSCH_2 - CH_2SH$

Diethylendisulfide



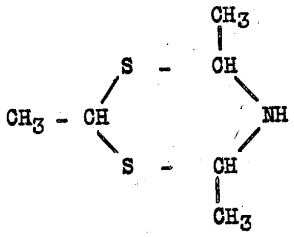
Diethylentetrasulfide



Ethylsulfide[3] $(C_2H_5 - SH)$

The following nitrogen-sulphur compounds were also prepared and tested:

Thialdine

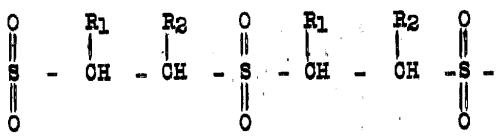


Carbothialdine $(C_5H_{10}N_2S_2)$

It appeared that trithioacetaldehyde and trithioacetone produced identical favorable effects. The other cyclic sulphur compounds showed no effect and still others only a weak effect. A 3:7-dilution of thialdine with cracked benzine produced a mixture with usable ignition-delay time but it proved rather unstable; precipitation began after about two months. Thialdine, however, was readily obtained by combining adequate quantities of ammonia, acetaldehyde ($CH_3 - CHO$), and hydrogen sulfide. It settles directly as an aqueous solution.

13. In fall 1948, the Special Technical Office was urged to develop triethylamine ($(C_2H_5)_3N$) from ethyl bromide (C_2H_5Br), and other amines on the basis of halogen-hydrocarbons. Ethyl bromide and ammonia was converted into triethylamine in an autoclave and produced the desired results. The remaining problem of determining the most favorable conditions was also solved successfully. Monoallylamine ($CH_2 = CH-CH_2-NH_2$), diallylamine, and triallylamine were also produced in a similar manner from allylchloride ($CH_2 = CH-CH_2Cl$). The hypergolic qualities of the allylamines were better than those of the corresponding ethylamines. Tetraethylethylendiamine (also called T 4) $[(C_2H_5)_2 = N-CH_2-CH_2-N = (C_2H_5)_2]$, which was synthesized from ethyl bromide and ethylendiamine ($H_2N-CH_2-CH_2-NH_2$), proved particularly effective. Work in this direction was later discontinued because the USSR allegedly lacked the raw materials required as basic elements for allyl compounds.

14. Other experimental work for preparing rocket propellants carried out on a minor scale, for example, was done with alkali-naphthalene compounds containing about one per cent sodium which also have ignition (hypergolic?) qualities with nitric acid. Since the substances applied proved too water-sensitive, this research work was discontinued. A catalytic hydrogenation process converting furfural (C_4H_3O CHO) into furfuryl alcohol (C_4H_3O . CH_2OH) followed. Also produced were sulfones which could be synthesized with sulphur dioxide on the basis of olefins, such as cyclohexane (C_2H_4),[4] butylene (C_4H_8), propylene (C_3H_6), and ethylene (C_2H_4). They were polymeric substances and were used as raw materials for varnish, depending on the length of the hydrocarbon chain



15. When benzines were used as diluting agents for propellant mixtures, the saturated paraffin hydrocarbons proved far inferior dissolvents than the aromatic benzines. Best suited were light benzines which, together with aromatic substances, showed a high percentage (up to five percent) of diolefins, such as cracked benzines. The Special Technical Office worked out a suitable analyzing process for this purpose. The benzine to be tested was analyzed into minute fractions by rectifying distillation and their titer of paraffin hydrocarbons, aromatics, and olefins were determined on the basis of their sulphuric acid adsorption and titer of iodine. The exact titer of benzol, toluol, highgrade aromatics, paraffins, olefins, and diolefins of about 20 kinds of cracked benzine was determined in this manner.

16. In 1949 some of the German specialists at the OKA Plant started working in the field of inhibitors. In an effort to reduce the corrosive effect of nitric acid on steel containers, small quantities of sulphonic acids, 0.05 to 0.5 percent, were added; they retarded corrosion considerably. This test was made on small steel plates, whose decrease in weight after lying in nitric acid for specified periods was to indicate the degree of corrosion. Sulphonic acids used as inhibitors included methionic acid ($CH_2(SO_3H)_2$), methantrisulfonic acid ($CH(SO_3H)_3$), and benzoldisulphonic acid ($C_6H_4(SO_3H)_2$). It appeared that the inhibiting effect was dependent on the number of sulphonic groups, which indicated that trisulphonic acids were particularly suited.

17. Between one and three kg of the products belonging to the field of rocket propelling agents, which had been prepared at the Special Technical Office of the OKA Plant and had been found suitable, were forwarded periodically to Chemical Plant No 94 in Moscow to be tested there. This plant had a special test chamber for these tests. Other major batches sent there included about 200 kg of a mixture composed of 60 percent (weight percentage) acetonine, 36 percent cracked benzol /benzine?/, and four percent butyrate of iron, in summer 1948; about 200 kg consisting of 40 percent acetonine, ten percent trithioacetone, and 50 percent cracked benzine (boiling range between 30 and 250 degrees centigrade), in summer 1949; about 200 kg of a mixture composed of 24 percent of acetonine, 12 percent aniline, and 60 percent cracked benzine with four percent dissolved elementary sulphur, in spring 1950; and about 100 kg of trithioacetone, of which a mixture was to be prepared in Moscow, in fall 1950. Large quantities of nitric acid mixed with 0.5 percent of iron were also shipped. The German specialists received no information on the results of the tests obtained in Moscow.

18. Since fall 1950, the technical school adjoining the laboratory of the OKA Plant had been trying, under pressure, a synthesis of acetonines in a special apparatus; obtaining, however, large quantities (up to 60 percent) of mesityl oxide, $(CH_3)_2 C = CH-CO-CH_3$. These experiments were still under way in May 1951.

19. Beginning in October 1950, all German specialists at the OKA Plant, except one, were assigned work in fields other than that of fuel agents. For example, a method for preparing methacrylnitril ($CH_2 = C - (CH_3) \cdot CN$) was worked out for the Rulon Plant, north of the OKA Plant, and Soviet personnel were given pertinent instruction. Isobutylene ($CH_3 - (CH)_2 - CH_3$),[5] about four to six percent of the total fraction, was extracted with 60 percent sulphuric acid from a butylene-propyene fraction of crack gases, and the sulfo-acid was hydrolized. The tertiary isobutyl alcohol ($(CH_3)_3 C - OH$) obtained therefrom was then purified by distillation. Pure isobutylene was recovered by means of sulphuric acid, and chlorided, and the chloride obtained was mixed with ammonia to form amine. The last stage of this process was oxidizing the amine to methacrylnitril with a silver contact. This process performed satisfactorily and showed the values calculated.

20. The German specialists had no access to production methods of the OKA Plant. They only knew that diphenylguanidine ($HN = C (NH - C_6H_5)_2$) was produced in one of the factories as a vulcanization accelerator for the rubber industry. This manufacture had been in operation for many years on the basis of a patent dating from 1935.

21. In 1949 and 1950, Soviet chemists made unsuccessful experiments at the operation laboratory to replace lead acetate or lead oxide (PbO) by iron salts for desulphurating the basic substance, that is, diphenylthio-urea (C_6H_5 - NH - CS - NH - C_6H_5). This research was subsequently assigned to one of the German chemists who, by virtue of his experiments, suggested pulverizing the used lead slag because it proved almost ineffective in its granular shape. The solvent was to be methyl alcohol (CH_3 - OH) instead of the hitherto used ethyl alcohol (C_2H_5 - OH), since the former would considerably increase production output with the same installation because of its better dissolving qualities. Subsequent research was continued by Soviet personnel.

22. Raw materials used in the laboratory and in the manufacturing plants of the OKA Plant included 94 percent pure ethyl alcohol, ethyl alcohol obtained from waste solutions of sulfite cellulose, methyl alcohol, ethylene ($CH_2 = CH_2$), ammonia, nitric acid, sulphuric acid, elementary sulphur, various metallic salts and metal oxides, mercury, and other chemicals generally used in laboratories. 25X1

[redacted]

2. [redacted] Comment: NH_4HS is the formula for ammonium hydrosulfide, not ammonium sulfide as stated in the report. The formula for ammonium sulfide is $(NH_4)_2$ S. 25X1

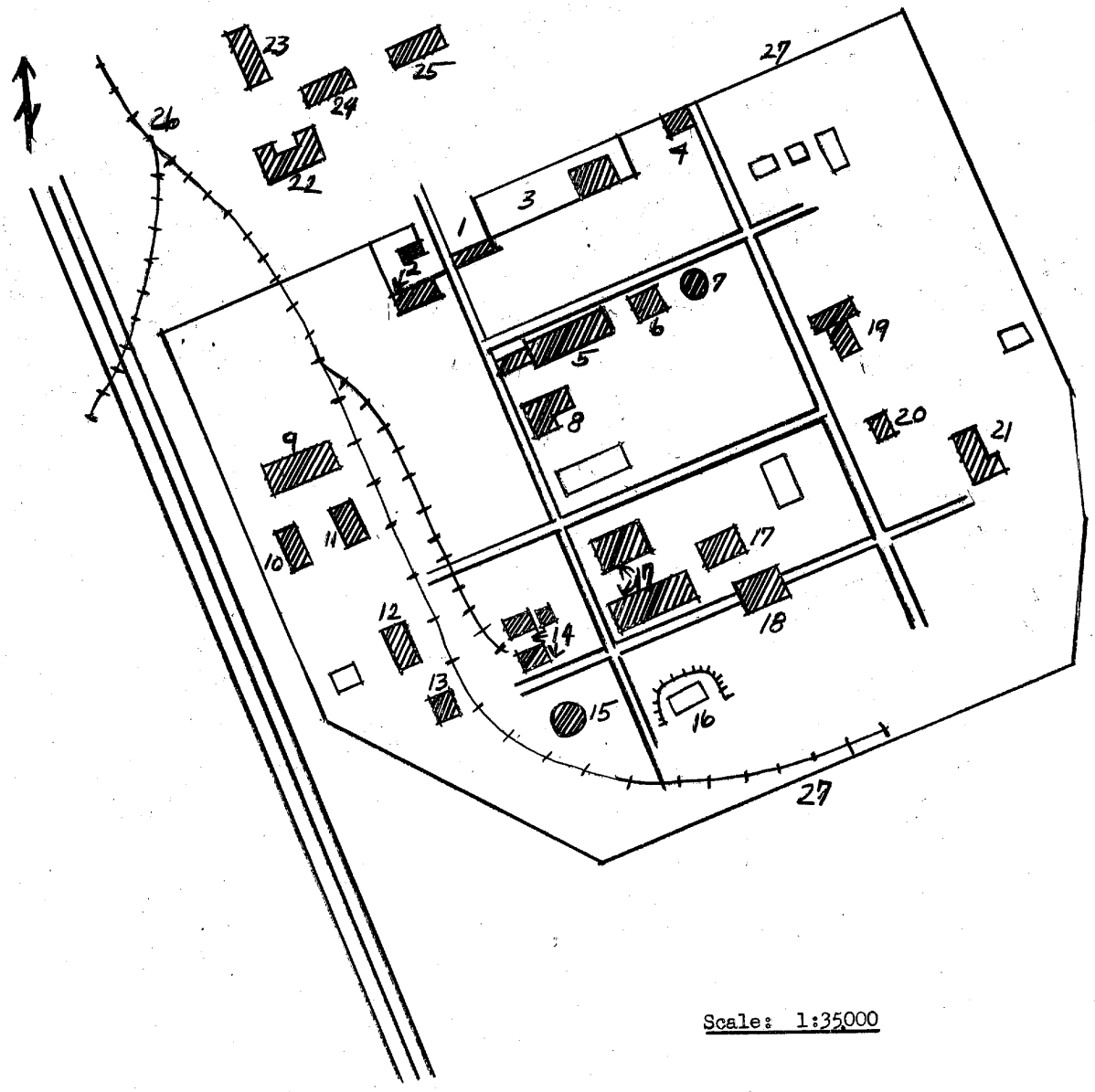
3. [redacted] Comment: The formula for ethylsulfide is $(C_2H_5)_2$ S, not C_2H_5 -SH as is given in the report, which is the formula for ethyl mercapton. 25X1

4. [redacted] Comment: Cyclohexane is not an olefin. 25X1

5. [redacted] Comment: $CH_3 - CH_2 - CH_3$ is the formula for n-propane and not isobutylene as stated in the report. The formula for isobutylene is $(CH_3)_2$ C = CH_2.

25X1

Layout Sketch of the OKA Chemical Plant in Dzerzhinsk



Scale: 1:35000

Legend

1. Guard building with passage and labor check point and, on the west, a thoroughfare for vehicles.
2. Four-story brick administration building.
3. Kennel for watchdogs.
4. Guard room with gate passage.
5. A four-story concrete building with an annex which served as a laboratory.
6. Technical institute.
7. Glassblower's workshop in a small round building.
8. Manufacturing shop for seed dressings equipped with can-filling devices.
9. A two-story manufacturing building with several tanks. A tube, about one meter long, projected from its roof. Soviet workers stated that ammonia gases were blown off there.
10. A building, designated as an acid storage, which was connected with other buildings of the plant by non-insulated piping. Workers wore rubber protective clothing in this building.
11. Concrete building with a spiral tube cooling system on its roof.
12. Manufacturing shop, a concrete building, around which a pungent smell of ammonia prevailed. Workers employed in the building wore protective clothing. Piping to the building extended from the building mentioned in item 11 and to the ammonia storage building mentioned in item 10.
13. A concrete factory building housing tanks and measuring instruments. Engine noises were heard outside the building.
14. Storage buildings. The northern one stored ammonia, and the southern one stored ethyl alcohol and sulphite spirits. These fluids arrived either in tank cars on the nearby railroad track and were tapped through pipes or arrived in drums. Methanol was also stored in the southern building in iron containers, each ten meters long and two meters in diameter. Three horizontal containers, each about 11 meters long and one meter in diameter, stood in the open on the east side of the northern building.
15. A gasholder-like container, about eight meters in diameter; was still under construction in July 1949.
16. A small storehouse, three sides of which were banked up.
17. New buildings of different sizes with a number of rooms which had tiled walls and flagged floors. The interior arrangement was not completed in September 1949.
18. Storage building for finished products, contained in demijohns, metal or cardboard buckets.
19. Mechanical workshop, a concrete building.
20. Carpenter's and cooper's shop.
21. New concrete building.

22. Club and library building.

23. MVD house.

24. Fire-brigade station.

25. Workshop.

26. Railroad spur track leading to the Igumnovo railroad station.

27. Plant fencing: a double barbed-wire fence with several wooden watchtowers.

Buildings which could not be clearly identified are unnumbered on the layout sketch.

25X1

Next 1 Page(s) In Document Denied